

## What is claimed is:

- 1. An array of samples comprising a plurality of solid-forms of a single compound-of-interest, each sample comprising the compound-of-interest, wherein said compound-of-interest is a small molecule, and at least two samples comprise solid-forms of the compound-of-interest each of the two solid-forms having a different physical state from the other.
- 2. An array comprising at least 24 samples each sample comprising a 10 compound-of-interest and at least one component, wherein:
  - (a) an amount of the compound-of-interest in each sample is less than about 1 gram; and
  - (b) at least one of the samples comprises a solid-form of the compound-of-interest.

15

30

- 3. The array of claim 2, wherein the amount of the compound-of-interest in each sample is less than about 100 milligrams.
- 4. The array of claim 2, wherein the amount of the compound-of-interest in 20 each sample is less than about 100 micrograms.
  - 5. The array of claim 2, wherein the amount of the compound-of-interest in each sample is less than about 100 nanograms.
- 25 6. The array of claim 2, wherein one or more samples differ from one or more other samples with respect to at least one of:
  - (a) amount or concentration of the compound-of-interest;
  - (b) the physical state of the solid-form of the compound-of-interest;
  - (c) the identity of one or more of the components;
  - (d) amount or concentration of one or more of the components;
  - (e) a physical state of one or more of the components; or
  - (f) pH.
- 7. The array of claim 2, wherein the compound-of-interest is a pharmaceutical, 35 an alternative medicine, a dietary supplement, a nutraceutical, a sensory material, an

4 - 7

agrochemical, an active component of a consumer formulation, or an active component of an industrial formulation.

- 8. The array of claim 2, wherein the compound-of-interest is a pharmaceutical.
  - 9. The array of claim 8, wherein the pharmaceutical is a small molecule.
- The array of claim 8, wherein the pharmaceutical is an oligonucleotide, a polynucleotide, an oligonucleotide conjugate, a polynucleotide conjugate, a protein, a
   peptide, a peptidomimetic, or a polysaccharide.
- 11. The array of claim 2, wherein one or more of the components is an excipient, a solvent, a non-solvent, a salt, an acid, a base, a gas, a pharmaceutical, a dietary supplement, an alternative medicine, a nutraceutical, a sensory compound, an agrochemical, an active component of a consumer formulation, an active component of an industrial formulation, a crystallization additive, an additive that affects particle or crystal size, an additive that structurally stabilizes crystalline or amorphous solid-forms, an additive that dissolves solid-forms, an additive that inhibits crystallization or precipitation, an optically-active solvent, an optically-active reagent; or an optically-active catalyst.

20

- 12. The array of claim 2, wherein each sample has been processed under a set of processing parameters.
- 13. The array of claim 12, wherein the set of processing parameters comprises at 25 least one of:
  - (a) adjusting a value of temperature;
  - (b) adjusting a time;
  - (c) adjusting pH;
  - (d) adjusting amount or concentration of the compound-of-interest;
- 30 (e) adjusting amount or concentration of one or more of the components:
  - (f) adding one or more additional components:
  - (g) nucleation:
  - (h) precipitation; or
  - (i) controlling the evaporation of one or more of the components;
- 35 or a combination thereof.

15

- 14. The array of claim 2, wherein the solid-form of the compound-of-interest is amorphous or crystalline.
- The array of claim 14, wherein the amorphous or crystalline form of the
   compound-of-interest is a salt, hydrate, anhydrous, co-crystal, dehydrated hydrate, solvate, desolvated solvate, clathrate, or inclusion.
  - 16. The array of claim 2, comprising two or more different polymorphs of the compound-of-interest.
  - 17. The array of claim 2, comprising two or more crystalline forms, wherein at least two of the crystalline forms have a different crystal habit.
    - 18. The array of claim 2, comprising at least about 48 samples.
  - 19. The array of claim 2, comprising at least about 96 samples.
    - 20. The array of claim 2, comprising at least about 1,000 samples.
- 20 21. The array of claim 2, comprising at least about 10,000 samples.
  - 22. A method of preparing an array of multiple solid-forms of a compound-of-interest comprising:
- (a) preparing at least 24 samples each sample comprising the compound-ofinterest and at least one component, wherein an amount of the compound-ofinterest in each sample is less than about 1 gram; and
  - (b) processing at least 24 of the samples to generate and array comprising at least two solid-forms of the compound-of-interest.
- 30 23. The method of claim 22, wherein the amount of the compound-of-interest in each sample is less than about 100 milligrams.
  - 24. The method of claim 22, wherein the amount of the compound-of-interest in each sample is less than about 100 micrograms.

- 25. The method of claim 22, wherein the amount of the compound-of-interest in each sample is less than about 100 nanograms.
- The method of claim 22, wherein one or more of the processed samplesdiffer from one or more other processed samples with respect to at least one of:
  - (a) amount or concentration of the compound-of-interest;
  - (b) the physical state of the solid-form of the compound-of-interest;
  - (c) the identity of one or more of the components;
  - (d) amount or concentration of one or more of the components;
- 10 (e) a physical state of one or more of the components; or
  - (f) pH.
- 27. The method of claim 22, wherein one or more of the components is an excipient, a solvent, a non-solvent, a salt, an acid, a base, a gas, a pharmaceutical, a dietary supplement, an alternative medicine, a nutraceutical, a sensory compound, an agrochemical, an active component of a consumer formulation, an active component of an industrial formulation, a crystallization additive, an additive that affects particle or crystal size, an additive that structurally stabilizes crystalline or amorphous solid-forms, an additive that dissolves solid-forms, an additive that inhibits crystallization or precipitation, an optically-active solvent, an optically-active reagent, or an optically-active catalyst.
  - 28. The method of claim 22, wherein processing the samples comprises at least one of:
    - (a) adjusting a value of temperature;
- 25 (b) adjusting a time;
  - (c) adjusting pH;
  - (d) adjusting amount or concentration of the compound-of-interest;
  - (e) adjusting amount or concentration of one or more of the components;
  - (f) adding one or more additional components;
- 30 (g) nucleation;
  - (h) precipitation; or
  - (i) controlling the evaporation of one or more of the components; or a combination thereof.

- 29. The method of claim 22, wherein at least one solid-form of the compound-of-interest is amorphous or crystalline.
- 30. The method of claim 29, wherein the amorphous or crystalline form of the compound-of-interest is a salt, hydrate, anhydrous, co-crystal, dehydrated hydrate, solvate, desolvated solvate, clathrate, or inclusion.
  - 31. The method of claim 22, wherein the array comprises two or more different polymorphs of the compound-of-interest.
  - 32. The method of claim 22, wherein the array comprises two or more crystalline forms of the compound-of-interest, wherein at least two of the crystalline forms have a different crystal habit.
- 15 33. The method of claim 22, wherein the compound-of-interest is a pharmaceutical, an alternative medicine, a dietary supplement, a nutraceutical, a sensory material, an agrochemical, an active component of a consumer formulation, or an active component of an industrial formulations.
- 20 34. The method of claim 22, wherein the compound-of-interest is a pharmaceutical.
  - 35. The method of claim 34, wherein the pharmaceutical is a small molecule.
- 25 36. The method of claim 34, wherein the pharmaceutical is an oligonucleotide, a polynucleotide, an oligonucleotide conjugate, a polynucleotide conjugate, a protein, a peptide, a peptidomimetic, or a polysaccharide.
- 37. The method of claim 22, wherein at least about 1000 samples are processed 30 in parallel.
  - 38. The method of claim 22, wherein at least about 10,000 samples are processed in parallel.

39. comprising: A method of screening a plurality of solid-forms of a compound-of-interest,

(a)

(c)

preparing at least 24 samples each sample comprising the compound-ofinterest and one or more components, wherein an amount of the compoundof-interest in each sample is less than about 1 gram;

(b)

processing at least 24 of the samples to generate an array wherein at least two of the processed samples comprise a solid-form of the compound-of-interest; and

analyzing the processed samples to detect at least one solid-form.

10

- The method of claim 39, wherein the amount of the compound-of-interest in 40. each sample is less than about 100 milligrams.
- The method of claim 39, wherein the amount of the compound-of-interest in 41. 15 each sample is less than about 100 micrograms.
  - The method of claim 39, wherein the amount of the compound-of-interest in 42. each sample is less than about 100 nanograms.

20

The method of claim 39, wherein one or more of the processed samples differ from one or more other processed samples with respect to at least one of:

(a)

amount or concentration of the compound-of-interest;

the physical state of the solid-form of the compound-of-interest;

(b) (c)

- the identity of one or more of the components;
- amount or concentration of one or more of the components; (d)
- a physical state of one or more of the components; or (e)
- pH. (f)
- The method of claim 39, wherein the processed samples are analyzed to 44. 30 determine if the solid-form is amorphous or crystalline.
- The method of claim 44, wherein the processed samples are analyzed by 45. visual inspection, video-optical microscopy, image analysis, polarized light analysis, near field scanning optical microscopy, far field scanning optical microscopy, atomic-force 35 microscopy, or micro-thermal analysis.



The method of claim 39, further comprising analyzing the detected solidform by infrared spectroscopy, near infrared spectroscopy, Raman spectroscopy, NMR, xray diffraction, neutron diffraction, powder x-ray diffraction, light microscopy, second harmonic generation, or electron microscopy.

- The method of claim 39, further comprising analyzing the detected solid-47. form by differential scanning calorimetry or thermal gravimetric analysis.
- The method of claim 39, wherein the compound-of-interest is a 48. 10 pharmaceutical, an alternative medicine, a dietary supplement, a nutraceutical, a sensory material, an agrochemical, an active component of a consumer formulation, or an active component of an industrial formulation.
- The method of claim 39, wherein one or more of the components is an 49. 15 excipient, a solvent, a non-solvent, a salt, an acid, a base, a gas, a pharmaceutical, a dietary supplement, an alternative medicine, a nutraceutical, a sensory compound, an agrochemical, an active component of a consumer formulation, an active component of an industrial formulation, a crystallization additive, an additive that affects particle or crystal size, an additive that structurally stabilizes crystalline or amorphous solid-forms, an additive that dissolves solid-forms, an additive that inhibits crystallization or precipitation, an opticallyactive solvent, an optically-active reagent, or an optically-active catalyst.
  - The method of claim 39 wherein processing the samples comprises at least 50. one of:
- adjusting a value of temperature; 25 (a)
  - adjusting a time; (b)
  - adjusting pH; (c)
  - adjusting amount or concentration of the compound-of-interest; (d)
  - adjusting amount or concentration of one or more of the components; (e)
- adding one or more additional components; 30 (f)
  - (g) nucleation;
  - precipitation; or (h)
  - controlling the evaporation of one or more of the components; or a combination thereof.

- 51. The method of claim 39, wherein at least one solid-form of the compound-of-interest is amorphous or crystalline.
- 52. The method of claim 51, wherein the amorphous or crystalline form of the compound-of-interest is a salt, hydrate, anhydrous, co-crystal, dehydrated hydrate, solvate, desolvated solvate, clathrate, or inclusion.
- 53. The method of claim 39, wherein the array comprises two or more different polymorphs of the compound-of-interest.
- 54. The method of claim 39, wherein the array comprises two or more crystalline forms of the compound-of-interest, wherein at least two of the crystalline forms have a different crystal habit.
- 15 55. The method of claim 39, wherein the compound-of-interest is a pharmaceutical.
  - 56. The method of claim 55, wherein the pharmaceutical is a small molecule.
- 20 57. The method of claim 55, wherein the pharmaceutical is an oligonucleotide, a polynucleotide, an oligonucleotide conjugate, a polynucleotide conjugate, a protein, a peptide, a peptidomimetic, or a polysaccharide.
- 58. The method of claim 39, wherein at least about 1000 samples are analyzed in 25 parallel.
  - 59. The method of claim 39, wherein at least about 10,000 samples are analyzed in parallel.
- 30 60. A method of identifying optimal solid-forms of a compound-of-interest, comprising:
  - (a) selecting at least one solid-form of the compound-of-interest present in an array comprising at least 24 samples each sample comprising the compoundof-interest and at least one component, wherein an amount of the compoundof-interest in each sample is less than about 1 gram; and

- (b) analyzing the solid-form.
- 61. The method of claim 60, wherein the amount of the compound-of-interest in each sample is less than about 100 milligrams.
- 62. The method of claim 60, wherein the amount of the compound-of-interest in each sample is less than about 100 micrograms.
- 63. The method of claim 60, wherein the amount of the compound-of-interest in 10 each sample is less than about 100 nanograms.
  - 64. The method of claim 60, wherein the optimal solid-forms have a large surface-to-volume ratio.
- 15 65. The method of claim 60, wherein one or more of the samples differ from one or more other samples with respect to at least one of:
  - (a) amount or concentration of the compound-of-interest;
  - (b) the physical state of the solid-form of the compound-of-interest;
  - (c) the identity of one or more of the components;
- 20 (d) amount or concentration of one or more of the components;
  - (e) a physical state of one or more of the components; or
  - (f) pH.
- 66. The method of claim 60, wherein the solid-form of the compound-of-interest is amorphous or crystalline.
  - 67. The method of claim 66, wherein the amorphous or crystalline form of the compound-of-interest is a salt, hydrate, anhydrous, co-crystal, dehydrated hydrate, solvate, desolvated solvate, clathrate, or inclusion.
  - 68. The method of claim 60, wherein the array comprises two or more different polymorphs of the compound-of-interest.
- 69. The method of claim 60, wherein the array comprises two or more crystalline forms, wherein the crystalline forms have a different crystal habit.

- 70. The method of claim 60, wherein the solid-form is analyzed by infrared spectroscopy, near infrared spectroscopy, Raman spectroscopy, NMR, x-ray diffraction, neutron diffraction, powder x-ray diffraction, light microscopy, electron microscopy, second harmonic generation, differential scanning calorimetry, or thermal gravimetric analysis.
- 71. The method of claim 60, wherein the solid-form is analyzed by an *in vitro* assay.
- 72. The method of claim 60, wherein one or more of the components is an excipient, a solvent, a non-solvent, a salt, an acid, a base, a gas, a pharmaceutical, a dietary supplement, an alternative medicine, a nutraceutical, a sensory compound, an agrochemical, an active component of a consumer formulation, an active component of an industrial formulation, a crystallization additive, an additive that affects particle or crystal size, an additive that structurally stabilizes crystalline or amorphous solid-forms, an additive that dissolves solid-forms, an additive that inhibits crystallization or precipitation, an optically-active solvent, an optically-active reagent, or an optically-active catalyst.
- 73. The method of claim 60, wherein the compound-of-interest is a pharmaceutical, an alternative medicine, a dietary supplement, a nutraceutical, a sensory
   20 material, an agrochemical, an active component of a consumer formulation, or an active component of an industrial formulation.
  - 74. The method of claim 60, wherein each sample in the array has been processed under a set of processing parameters.

- 75. The method of claim 74, wherein the set of processing parameters comprises at least one of:
  - (a) adjusting a value of temperature;
  - (b) adjusting a time;
- 30 (c) adjusting pH;
  - (d) adjusting amount or concentration of the compound-of-interest;
  - (e) adjusting amount or concentration of one or more of the components;
  - (f) adding one or more additional components;
  - (g) nucleation;
- 35 (h) precipitation; or

- (i) controlling the evaporation of one or more of the components; or a combination thereof.
- 76. The method of claim 60, wherein the array comprises two or more different polymorphs of the compound-of-interest.
  - 77. The method of claim 60, wherein the array comprises two or more crystalline forms of the compound-of-interest, wherein at least two of the crystalline forms have a different crystal habit.
  - 78. The method of claim 60, wherein the compound-of-interest is a pharmaceutical.
    - 79. The method of claim 78, wherein the pharmaceutical is a small molecule.
- 80. The method of claim 78, wherein the pharmaceutical is an oligonucleotide, a polynucleotide, an oligonucleotide conjugate, a polynucleotide conjugate, a protein, a peptide, a peptidomimetic, or a polysaccharide.
- 20 81. The method of claim 60, wherein the array comprises at least 48 samples.
  - 82. The method of claim 60, wherein the array comprises at least 96 samples.
- 83. The method of claim 60, wherein at least about 10 solid-forms are analyzed 25 in parallel.
  - 84. The method of claim 60, wherein at least about 100 solid-forms are analyzed in parallel.
- 30 85. The method of claim 60, wherein at least about 1,000 solid-forms are analyzed in parallel.
  - 86. A method to determine sets of conditions and/or components to produce particular solid-forms of a compound-of-interest, comprising:

- (a) preparing at least 24 samples each sample comprising the compound-of-interest and one or more components, wherein an amount of the compound-of-interest in each sample is less than about 1 gram;
- (b) processing at least 24 of the samples to generate an array wherein at least one of the processed samples comprises a solid-form of the compound-ofinterest; and
  - (c) selecting samples having the solid-forms in order to identify the sets of conditions and/or components.
- 10 87. The method of claim 86, wherein the amount of the compound-of-interest in each sample is less than about 100 milligrams.
  - 88. The method of claim 86, wherein the amount of the compound-of-interest in each sample is less than about 100 micrograms.

5

- 89. The method of claim 86, wherein the amount of the compound-of-interest in each sample is less than about 100 nanograms.
- 90. The method of claim 86, wherein the desired solid-form has a large surface-20 to-volume ratio.
  - 91. The method of claim 86, wherein one or more of the processed samples differ from one or more other processed samples with respect to at least one of:
    - (a) amount or concentration of the compound-of-interest;
- 25 (b) the physical state of the solid-form of the compound-of-interest;
  - (c) the identity of one or more of the components;
  - (d) amount or concentration of one or more of the components;
  - (e) a physical state of one or more of the components; or
  - (f) pH.

- 92. The method of claim 86, wherein processing the samples comprises at least one of:
  - (a) adjusting a value of temperature;
  - (b) adjusting a time;
- 35 (c) adjusting pH;

- (d) adjusting amount or concentration of the compound-of-interest;
- (e) adjusting amount or concentration of one or more of the components;
- (f) adding one or more additional components;
- (g) nucleation;
- 5 (h) precipitation; or
  - (i) controlling the evaporation of one or more of the components; or a combination thereof.
- 93. The method of claim 86, wherein at least one solid-form of the compound-10 of-interest is amorphous or crystalline.
  - 94. The method of claim 93, wherein the amorphous or crystalline form of the compound-of-interest is a salt, hydrate, anhydrous, co-crystal, dehydrated hydrate, solvate, desolvated solvate, clathrate, or inclusion.
  - 95. The method of claim 86, wherein the array comprises two or more different polymorphs of the compound-of-interest.
- 96. The method of claim 86, wherein the array comprises two or more crystalline forms of the compound-of-interest, wherein at least two of the crystalline forms have a different crystal habit.
- 97. The method of claim 86, wherein the compound-of-interest is a pharmaceutical, an alternative medicine, a dietary supplement, a nutraceutical, a sensory
  25 material, an agrochemical, an active component of a consumer formulation, or an active component of an industrial formulation.
- 98. The method of claim 86, wherein one or more of the components is an excipient, a solvent, a non-solvent, a salt, an acid, a base, a gas, a pharmaceutical, a dietary supplement, an alternative medicine, a nutraceutical, a sensory compound, an agrochemical, an active component of a consumer formulation, an active component of an industrial formulation, a crystallization additive, an additive that affects particle or crystal size, an additive that structurally stabilizes crystalline or amorphous solid-forms, an additive that dissolves solid-forms, an additive that inhibits crystallization or precipitation, an optically-active solvent, an optically-active reagent, or an optically-active catalyst.

- 99. The method of claim 86, wherein the compound-of-interest is a pharmaceutical.
  - 100. The method of claim 99, wherein the pharmaceutical is a small molecule.

101. The method of claim 99, wherein the pharmaceutical is an oligonucleotide, a polynucleotide, an oligonucleotide conjugate, a polynucleotide conjugate, a protein, a peptide, a peptidomimetic, or a polysaccharide.

- 10 102. The method of claim 86, wherein at least about 1000 samples are processed in parallel.
  - 103. The method of claim 86, wherein at least about 10,000 samples are processed in parallel.
- 15
  104. A method of screening conditions and/or components for compatibility with one or more selected solid-forms of a compound-of-interest, comprising:
  - (a) preparing at least 24 samples each sample comprising the compound-of-interest in solid or dissolved form and one or more components, wherein an amount of the compound-of-interest in each sample is less than about 1
    - (b) processing at least 24 of the samples to generate an array of said selected solid-forms; and
    - (c) analyzing the array.
  - 105. The method of claim 104, wherein the amount of the compound-of-interest in each sample is less than about 100 milligrams.
- 106. The method of claim 104, wherein the amount of the compound-of-interest 30 in each sample is less than about 100 micrograms.
  - 107. The method of claim 104, wherein the amount of the compound-of-interest in each sample is less than about 100 nanograms.

20

- The method of claim 104, wherein one or more of the processed samples 108. differ from one or more other processed samples with respect to at least one of:
  - amount or concentration of the compound-of-interest; (a)
  - the identity of one or more of the components; (b)
- amount or concentration of one or more of the components; 5 (c)
  - a physical state of one or more of the components; or (d)
  - pH. (e)
  - The method of claim 104, wherein processing the samples comprises at least 109.
- 10 one of:
  - adjusting a value of temperature; (a)
  - adjusting a time; (b)
  - adjusting pH; (c)
  - adjusting amount or concentration of the compound-of-interest; (d)
- adjusting amount or concentration of one or more of the components; 15 (e)
  - adding one or more additional components; (f)
  - nucleation: (g)
  - precipitation; or (h)
  - controlling the evaporation of one or more of the components; (i)
- 20 or a combination thereof.
  - The method of claim 104, wherein the selected solid form of the compound-110. of-interest is a salt, a hydrate, a co-crystal, a dehydrated hydrate, a solvate, a desolvated solvate, a clathrate, an inclusion, a particular polymorph, or of a particular crystal habit.
- 25
- The method of claim 104, wherein the compound-of-interest is a 111. pharmaceutical, an alternative medicine, a dietary supplement, a nutraceutical, a sensory material, an agrochemical, an active component of a consumer formulation, or an active component of an industrial formulation.
- 30
- The method of claim 104, wherein one or more of the components is an 112. excipient, a solvent, a non-solvent, a salt, an acid, a base, a gas, a pharmaceutical, a dietary supplement, an alternative medicine, a nutraceutical, a sensory compound, an agrochemical, an active component of a consumer formulation, an active component of an industrial
- 35 formulation, a crystallization additive, an additive that affects particle or crystal size, an

additive that structurally stabilizes crystalline or amorphous solid-forms, an additive that dissolves solid-forms, or an additive that inhibits crystallization or precipitation.

- 113. The method of claim 104, wherein the compound-of-interest is a 5 pharmaceutical.
  - 114. The method of claim 113, wherein the pharmaceutical is a small molecule.
- The method of claim 113, wherein the pharmaceutical is an oligonucleotide,
  a polynucleotide, an oligonucleotide conjugate, a polynucleotide conjugate, a protein, a
  peptide, a peptidomimetic, or a polysaccharide.
  - 116. The method of claim 104, wherein at least about 1000 samples are processed in parallel.
  - 117. The method of claim 104, wherein at least about 10,000 samples are processed in parallel.
  - 118. A system to identify optimal solid-forms of a compound-of-interest,
- 20 comprising:

- (a) an automated distribution mechanism effective to prepare at least 24 samples, each sample comprising the compound-of-interest and one or more components, wherein an amount of the compound-of-interest in each sample is less than about 1 gram;
- 25 (b) an system effective to process the samples to generate an array comprising at least one solid-form of the compound-of-interest; and
  - (c) a detector to detect the solid-form.
- 119. The system of claim 118, wherein the amount of the compound-of-interest in 30 each sample is less than about 100 milligrams.
  - 120. The system of claim 118, wherein the amount of the compound-of-interest in each sample is less than about 100 micrograms.

- 121. The system of claim 118, wherein the amount of the compound-of-interest in each sample is less than about 100 nanograms.
- 122. The system of claim 118, wherein the optimal solid-forms have a large 5 surface-to-volume ratio.
  - 123. The system of claim 118, wherein the automated distribution mechanism is effective to deliver and the detector is effective to detect nanogram quantities of the compound-of-interest.
  - 124. The system of claim 118, wherein the detector is a video optical microscope, an image analyzer, an optical microscope, or a polarimeter.
- 125. The system of claim 118, further comprising an analyzer to analyze the 15 detected solid-form.
- 126. The system of claim 125, wherein the analyzer is an infrared spectrophotometer, a second harmonic generation optical spectrometer, a mass spectrometer, a nuclear magnetic resonance spectrometer, a near infrared
  20 spectrophotometer, a Raman spectrophotometer, an x-ray powder diffractometer, a differential scanning calorimeter, a thermal gravimetric analyzer, a light microscope, or an electron microscope.
  - 127. The system of claim 125, wherein the analyzer is an in vitro assay.
  - 128. A method to determine a set of processing parameters and/or components to inhibit the formation of a solid-form of a compound-of-interest, comprising:
    - (a) preparing at least 24 samples each sample comprising a solution of the compound-of-interest and one or more components, wherein an amount of the compound-of-interest in each sample is less than about 1 gram;
    - (b) processing at least 24 of the samples under a set of processing parameters; and
    - (c) selecting the processed samples not having the solid-form to identify the set of processing parameters and/or components.

- 98 -

35

30

- 129. The method of claim 128, wherein the amount of the compound-of-interest in each sample is less than about 100 milligrams.
- 130. The method of claim 128, wherein the amount of the compound-of-interest in each sample is less than about 100 micrograms.
  - 131. The method of claim 128, wherein the amount of the compound-of-interest in each sample is less than about 100 nanograms.
- 10 132. The method of claim 128, wherein one or more of the processed samples differ from one or more other processed samples with respect to at least one of:
  - (a) amount or concentration of the compound-of-interest;
  - (b) the identity of one or more of the components;
  - (c) amount or concentration of one or more of the components;
- 15 (d) a physical state of one or more of the components; or
  - (e) pH.
  - 133. The method of claim 128, wherein processing the samples comprises at least one of:
- 20 (a) adjusting a value of temperature;
  - (b) adjusting a time;
  - (c) adjusting pH;
  - (d) adjusting amount or concentration of the compound-of-interest;
  - (e) adjusting amount or concentration of one or more of the components;
- 25 (f) adding one or more additional components;
  - (g) nucleation;
    - (h) precipitation; or
    - (i) controlling the evaporation of one or more of the components;

or a combination thereof.

- 134. The method of claim 128, wherein one or more of the components is an excipient, a solvent, a non-solvent, a salt, an acid, a base, a gas, a pharmaceutical, a dietary supplement, an alternative medicine, a nutraceutical, a sensory compound, an agrochemical, an active component of a consumer formulation, an active component of an industrial
- 35 formulation, a crystallization additive, an additive that affects particle or crystal size, an

30

additive that structurally stabilizes crystalline or amorphous solid-forms, an additive that dissolves solid-forms, an additive that inhibits crystallization or precipitation, an optically-active solvent, an optically-active reagent, or an optically-active catalyst.

- 5 135. The method of claim 128, wherein the compound-of-interest is a pharmaceutical, an alternative medicine, a dietary supplement, a nutraceutical, or an agrochemical.
- 136. The method of claim 128, wherein the compound-of-interest is a 10 pharmaceutical.
  - 137. The method of claim 136, wherein the pharmaceutical is a small molecule.
- 138. The method of claim 136, wherein the pharmaceutical is an oligonucleotide, a polynucleotide, an oligonucleotide conjugate, a polynucleotide conjugate, a protein, a peptide, a peptidomimetic, or a polysaccharide.
  - 139. The method of claim 128, wherein at least about 1000 samples are processed in parallel.
  - 140. The method of claim 128, wherein at least about 10,000 samples are processed in parallel.
- 141. A method to determine a set of processing parameters and/or components to dissolve or partially dissolve a solid-form of a compound-of-interest, comprising:
  - (a) preparing at least 24 samples each sample comprising a solid-form of the compound-of-interest and one or more components, wherein an amount of the compound-of-interest in each sample is less than about 1 gram;
  - (b) processing at least 24 of the samples under a set of processing parameters; and
    - (c) selecting the processed samples wherein the solid-form dissolved or partially dissolved to identify the set of processing parameters and/or components.
- 142. The method of claim 141, wherein the amount of the compound-of-interest 35 in each sample is less than about 100 milligrams.

- 143. The method of claim 141, wherein the amount of the compound-of-interest in each sample is less than about 100 micrograms.
- 144. The method of claim 141, wherein the amount of the compound-of-interestin each sample is less than about 100 nanograms.
  - 145. The method of claim 141, wherein one or more of the processed samples differ from one or more other processed samples with respect to at least one of:
    - (a) amount or concentration of the compound-of-interest;
- 10 (b) the physical state of the compound-of-interest;
  - (c) the identity of one or more of the components;
  - (d) amount or concentration of one or more of the components;
  - (e) a physical state of one or more of the components; or
  - (f) pH.

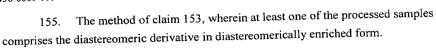
- 146. The method of claim 141, wherein processing the samples comprises at least one of:
  - (a) adjusting a value of temperature;
  - (b) adjusting a time;
- 20 (c) adjusting pH;
  - (d) adjusting amount or concentration of the compound-of-interest;
  - (e) adjusting amount or concentration of one or more of the components;
  - (f) adding one or more additional components;
  - (g) nucleation;
- 25 (h) precipitation; or
  - (i) controlling the evaporation of one or more of the components; or a combination thereof.
- 147. The method of claim 141, wherein one or more of the components is an excipient, a solvent, a non-solvent, a salt, an acid, a base, a gas, a pharmaceutical, a dietary supplement, an alternative medicine, a nutraceutical, a sensory compound, an agrochemical, an active component of a consumer formulation, an active component of an industrial formulation, a crystallization additive, an additive that affects particle or crystal size, an additive that structurally stabilizes crystalline or amorphous solid-forms, an additive that

25

30

dissolves solid-forms, an additive that inhibits crystallization or precipitation, an optically-active solvent, an optically-active reagent, or an optically-active catalyst.

- The method of claim 141, wherein the compound-of-interest is a
   pharmaceutical, an alternative medicines, a dietary supplement, a nutraceutical, or an agrochemical.
  - 149. The method of claim 141, wherein the compound-of-interest is a pharmaceutical.
    - 150. The method of claim 149, wherein the pharmaceutical is a small molecule.
- 151. The method of claim 149, wherein the pharmaceutical is an oligonucleotide, a polynucleotide, an oligonucleotide conjugate, a polynucleotide conjugate, a protein, a peptide, a peptidomimetic, or a polysaccharide.
  - 152. The method of claim 141, wherein at least about 10,000 samples are processed in parallel.
- 20 153. A method for determining conditions and/or components which produce a compound-of-interest or a diastereomeric derivative thereof in stereomerically enriched or conglomerate form, comprising:
  - (a) preparing at least 24 samples each sample comprising the compound-ofinterest or a diastereomeric derivative thereof and one or more components, wherein an amount of the compound-of-interest or the diastereomeric derivative in each sample is less than about 1 gram;
  - (b) processing at least 24 of the samples to generate an array wherein at least one of the processed samples comprises the compound-of-interest or the diastereomeric derivative in stereomerically enriched or conglomerate form; and
  - (c) selecting the stereomerically enriched or conglomerate samples in order to identify the set of conditions and/or components.
- 154. The method of claim 153, wherein at least one of the processed samples comprises the compound-of-interest in enantiomerically enriched form.



- 156. The method of claim 153, wherein the amount of the compound-of-interest or the diastereomeric derivative in each sample is less than about 100 milligrams.
  - 157. The method of claim 153, wherein the amount of the compound-of-interest or the diastereomeric derivative in each sample is less than about 100 micrograms.
- 10 158. The method of claim 153, wherein the amount of the compound-of-interest or the diastereomeric derivative in each sample is less than about 100 nanograms.
  - 159. The method of claim 153, wherein one or more of the processed samples differ from one or more other processed samples with respect to at least one of:
- 15 (a) amount or concentration of the compound-of-interest or the diastereomeric derivative;
  - (b) the identity of the diastereomeric derivative;
  - (c) the physical state of the solid-form of the compound-of-interest or the diastereomeric derivative;
- 20 (d) the identity of one or more of the components;
  - (e) amount or concentration of one or more of the components;
  - (f) a physical state of one or more of the components; or
  - (g) pH.
- 25 160. The method of claim 153, wherein processing the samples comprises at least one of:
  - (a) adjusting a value of temperature;
  - (b) adjusting a time;
  - (c) adjusting pH;
- 30 (d) adjusting amount or concentration of the compound-of-interest or the diastereomeric derivative;
  - (e) adjusting amount or concentration of one or more of the components;
  - (f) adding one or more additional components;
  - (g) nucleation; or
- 35 (h) controlling the evaporation of one or more of the components;

## or a combination thereof.

- 161. The method of claim 153, wherein the compound-of-interest is a pharmaceutical, an alternative medicine, a dietary supplement, a nutraceutical, a sensory material, an agrochemical, an active component of a consumer formulation, or an active component of an industrial formulation.
- 162. The method of claim 153, wherein one or more of the components is an excipient, a solvent, a non-solvent, a salt, an acid, a base, a gas, a pharmaceutical, a dietary supplement, an alternative medicine, a nutraceutical, a sensory compound, an agrochemical, an active component of a consumer formulation, an active component of an industrial formulation, a crystallization additive, an additive that affects particle or crystal size, an additive that structurally stabilizes crystalline or amorphous solid-forms, an additive that dissolves solid-forms, an additive that inhibits crystallization or precipitation, an optically-active solvent, an optically-active reagent, or an optically-active catalyst.
  - 163. The method of claim 153, wherein the compound-of-interest is a pharmaceutical.
- 20 164. The method of claim 163, wherein the pharmaceutical is a small molecule.
  - 165. The method of claim 163, wherein the pharmaceutical is an oligonucleotide, a polynucleotide, an oligonucleotide conjugate, a polynucleotide conjugate, a protein, a peptide, a peptidomimetic, or a polysaccharide.
    - 166. The method of claim 153, wherein the array comprises at least 48 samples.
      - 167. The method of claim 153, wherein the array comprises at least 96 samples.
- 30 168. The method of claim 153, wherein at least about 1000 samples are processed in parallel.
  - 169. The method of claim 153, wherein at least about 10,000 samples are processed in parallel.

35